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Ultrasonic Velocity Technique for Monitoring Property Changes in Fiber-Reinforced Ceramic Matrix Composites

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SUMMARY

A novel technique for measuring ultrasonic velocity was used to monitor changes that occur during processing and heat treatment of a SiC/RBSN composite. Results indicated that correlations exist between the ultrasonic velocity data and elastic modulus and also interfacial shear strength data determined from mechanical tests. The ultrasonic velocity data can differentiate changes in axial modulus as well as in interfacial shear strength. The advantages and potential of this NDE method for fiber-reinforced ceramic matrix composite applications are discussed.

INTRODUCTION

The mechanical performance of fiber-reinforced ceramic matrix composites (FRCMC) depends upon fiber, fiber/matrix interface and matrix properties (ref. 1). Changes in component properties either during fabrication or service will influence mechanical properties. These changes are generally measured by destructive tests. However, it would be desirable to have a nondestructive (NDE) method for monitoring changes in these properties.

The purpose of the present work was to devise and use an ultrasonic velocity method to monitor changes in matrix properties and interfacial shear strength in a unidirectionally reinforced SiC/RBSN composite. Two modes of ultrasonic wave propagation were measured along the fiber direction for specimens prepared under various conditions of density, SiC fiber surface coating, and heat treatment history. Differences in velocity between these conditions were then compared to differences in mechanical properties as determined by destructive tests.

EXPERIMENTAL

Ultrasonic Set-up

A schematic of the ultrasonic velocity measurement unit is shown in figure 1. The unit employs two transducers. Pairs of broadband transducers with center frequencies of 0.5, 1.0, and 2.25 MHz are used. They are coupled to the same surface of the specimen and are separated by a distance S which is varied from 38.1 to 70 mm.

The transducers are coupled to the specimen through silicone rubber pads bonded to the transducer face.

The pads are 12.7 x 5 mm and about 2 mm thick when uncompressed. During measurement, the couplant pads are compressed onto the specimen with combined force of about 2.5 N. An advantage of the silicone pad coupling is that it allows ultrasonic measurement of porous specimens without leaving any residue. The sending transducer introduces ultrasound. The receiving transducer captures the signal after it has interacted with the specimen. These collected signals are digitized, stored, and analyzed.

Velocity Measurement Technique

The ultrasonic set-up described above is similar to the acousto-ultrasonic (AU) method (ref. 2). The AU method measures the amplitude and frequency distribution of ultrasonic signals that arrive at the receiving transducer. There are discrete pulse components in this AU signal that are relatively low in amplitude and frequency. In many cases these pulses are not detectable. However, under conditions where high frequencies are damped and ringdown is suppressed, the discrete pulses become evident. This damping and ringdown suppression is achieved through optimizing couplant pad pressure. The optimum combined force on the pair of transducers for our work is found to be approximately 2.5 N.

Figure 2 shows the time domain of a typical ultrasonic signal collected using two 2.25 MHz center frequency transducers. The signal displays two very prominent low frequency pulses. The pulse labeled 1 travels directly from the sender to the receiver along the distance, S. There is another pulse of interest in figure 2. It is the relatively high frequency pulse at the very beginning of the signal. It is labeled 2 in figure 2. This also travels directly the distance S.

By windowing the ultrasonic pulses 1 and 2 separately, one can determine their arrival times. In order to study these pulses more precisely, different transducer frequency ranges were employed. For pulse 1, two 0.5 MHz center frequency transducers were used. For pulse 2, two 1.0 MHz transducers were used.

Precise velocity values were determined from the ultrasonic pulses by varying the transducer spacing, S, shown in figure 1 from 38.1 to 70 mm for a total of seven spacings. For each spacing a waveform was collected. Each waveform was Fourier transformed and gaussian filtered to maximize the frequency range of the pulse of interest. The signal was then transformed back to the time domain where the time of the pulse arrival was determined.

With this set of seven transducer spacings, S_j, and arrival times, t_j, a velocity can be determined from the slope of a regression curve as illustrated in figure 3 (i.e., Velocity = $\Delta S/\Delta t$). The use of linear regression to determine velocity allows one to evaluate the accuracy of the data through the "goodness-of-fit" of the curve.

Based upon the relative magnitudes of the velocities and their general range of values (ref.3), pulse 1 is seen to behave as a shear wave and its velocity is designated as VS. Similarly, pulse 2 behaves as a longitudinal wave and is designated as VL.

The primary limit to this technique is the extent that the through length pulses can be resolved to yield arrival times. The initial experience is that the VS pulses are more resolvable and reproducible than VL pulses. This is because they are of larger amplitude.

Fabrication of Materials

The starting materials for SiC/RBSN composite fabrication were SiC monofilaments[1] of nominal diameter 140 μm and high purity silicon powder[2] of average particle size of 0.3 μm. Two types of SiC fibers were used; uncoated and carbon-coated. These fibers are designated as SCS-0 and SCS-6, respectively. The composites were fabricated by conventional ceramic fabrication methods using polymer fugitive binder. The composite was fabricated in a three-step process. In the first step, SCS-6 fiber mats and silicon powder tapes were prepared using polymer binders. For the standard SCS-6/RBSN composites fabrication, powder tape containing silicon and 2.5 percent NiO was used. For hot-isostatically pressed SiC/RBSN, a mixture of silicon, 2.5 percent NiO, and 5 percent MgC was used. In the second step, alternate layers of SiC fiber mats and silicon tapes were stacked in a metal die and hot pressed under appropriate applied pressure and temperature. This resulted in handleable SiC/Si preforms. In the third step, preforms were heated in high purity nitrogen between 1000 °C and 1400 °C for up to 100 hr to form standard grade SiC/RBSN composites. A more detailed description of the composite fabrication is given elsewhere (ref.4). The typical dimensions of the as-nitrided panels were 150 by 50 by 2.2 mm.

For hot-isostatically pressed SCS-6/RBSN composites, the consolidation and nitridation procedures were similar to that of the standard grade SCS-6/RBSN composites. However, the nitrided composites were further densified to full density by hot-isostatic pressing (HIPing) under conditions discussed previously (ref.5).

For fiber/matrix interface modification, two approaches were employed. In the first approach, SCS-0 fibers were substituted for SCS-6 in the standard grade SCS-6/RBSN composites. These composites displayed strong bonding between the fiber and matrix. In the second approach, the standard grade SCS-6/RBSN composite was heat treated in oxygen at 600 °C for 100 hr. This treatment caused oxidation of the carbon-rich surface coating on SiC fibers and resulted in loss of bonding between SiC fibers and RBSN matrix. In the present work, we examined at least two specimens under each testing condition.

Tensile Strength Measurements

As-fabricated composite panels were ground with diamond impregnated grinders to remove loose Si_3N_4 particles, scratches, and surface roughness. They were then sliced into tensile specimens using a diamond impregnated cutoff wheel. Nominal dimensions of the tensile specimens were 127 by 12.7 by 2.0 mm. The as-fabricated and heat treated specimens were prepared for tensile tests by adhesively bonding glass fiber reinforced epoxy tabs at their ends, leaving 50 mm as the test gauge length. A wire wound strain gauge was adhesively bonded to the specimen gauge section for monitoring axial strains. The tensile tests were conducted at room-temperature in a tensile testing machine[3] at a cross head speed of 1.3 mm/min. At each condition, at least three specimens were tested.

[1]Textron Specialty Materials, Lowell, Massachusetts.

[2]Union Carbide.

[3]Instron Corporation, Canton, Massachusetts.

Ultrasonic Velocity Measurements

Tensile specimens were subjected to four ultrasonic velocity runs for VL determination and four runs for VS. A run is a set of seven spacing and pulse arrivals that are regressed to obtain a velocity. Two runs were performed on each side of a given specimen. The average and standard deviation of the four values were used in assessing the sensitivity of velocity to mechanical strength in the specimens. Generally, the standard deviation for VS averages was about 2 percent. For VL the standard deviation was less than 10 percent unless VL on one side of the specimen was obviously a different value than that of the other. In this case VL was still averaged over the two sides for the purpose of comparison with mechanical properties. VS was always the same on both sides.

RESULTS

Mechanical Properties

The mechanical property data obtained from the stress-strain curves, the ultrasonic velocity data, and the interfacial shear strength data measured for the SiC/RBSN composites are shown in table I.

Typical room-temperature tensile stress-strain curves are shown in figure 4 for the standard SCS-6/RBSN and HIPed SCS-6/RBSN, the SCS-0/RBSN, and the standard SCS-6/RBSN composites heat treated in oxygen at 600 °C for 100 hr. The stress-strain curves of the composite specimens, except for the SCS-0/RBSN composite, show three distinct regions an initial linear region, followed by a nonlinear region, and then a second linear region. Previous studies (ref. 6) have indicated that matrix cracking normal to the fibers and generation of regular multiple matrix cracking led to the nonlinear region. The standard and HIPed SCS-6/RBSN composites displayed no strain capability beyond ultimate stress. The oxidized SCS-6/RBSN composite showed some strain capability beyond the ultimate stress, i.e. maximum stress. The stress-strain curve for SCS-0/RBSN composites had only an initial linear region and displayed no strain capability beyond matrix fracture.

The interfacial shear strength, ISS, was measured by the matrix crack spacing method from knowledge of the average matrix crack spacing, the first matrix cracking stress, and the elastic properties of the constituents. Details of the method can be found elsewhere (refs. 6 and 7). Since SCS-0/RBSN composites failed similar to monolithic ceramics, the matrix crack spacing method could not be used. The interfacial shear strength of SCS-0/RBSN composites was estimated from fiber push out results (ref.8).

The four fabrication conditions illustrated in figure 4 can be used to assess the validity of the assertion that the ultrasonic velocities, VL and VS, measured in this work are sensitive to changes in, respectively, axial tensile modulus (E) and interfacial shear strength (ISS) in SiC/RBSN composites.

Density Effects

Figure 5 shows the difference in mechanical properties between the standard SCS-6/RBSN specimens, with typical density of 2.40 g/cm^3 and HIP'ed specimens with density of 3.04 g/cm^3. Similarly, figure 6 shows the dependence of the ultrasonic velocities on density. Taking these two figures together, we see that the elastic modulus E, and the ultrasonic property VL both are increasing functions of specimen density. However, ISS and VS are essentially independent of density.

Interface Effects

Figures 7 and 8 compare the ultrasonic velocity data and the mechanical property data of the standard SCS-6/RBSN and the SCS-0/RBSN. Figure 7 shows that VS and ISS are both greater with the uncoated SCS-0 fibers. The ISS in SCS-0 is so large that it is beyond precise determination with the fiber push-out test. The lower bound is taken as 200 MPa. The difference of VS is not as extreme between the SCS-6/RBSN and SCS-0/RBSN, but it is statistically significant.

Figure 8 shows that VL and E have a similar trend with respect to type of fiber. Neither of these variables shows a statistical difference with the fiber type.

Figure 9 shows the room-temperature mechanical properties, E and ISS, for standard grade SCS-6/RBSN before and after 100 hr of oxidation treatment at 600 °C. Both mechanical properties are degraded by the oxidation heat treatment. Figure 10 shows that the measured ultrasonic velocities, VL and VS, are also degraded by the heat treatment.

DISCUSSION

In one theoretical model, the ultrasonic wave propagation in unidirectional fiber reinforced composites, the wave propagation is treated in terms of bulk waves (refs. 9 and 10). In another, it is treated in terms of Lamb waves (ref. 11). Both models predict the existence of wave modes that correlate with the axial modulus and also separate modes that correlate with shear modulus. The relationship between the ultrasonic modulus and ultrasonic velocity has been experimentally confirmed in boron fiber-reinforced aluminum matrix composites (ref. 12). The calculated ultrasonic modulus values of this system were in good agreement with the modulus values measured by mechanical property testing. However, no such relationship has been experimentally confirmed for shear properties of a composite. This is likely due to the difficulty of generating and detecting shear type waves in materials. Often, this requires special couplant materials as well as specimen geometry and surface preparation. However, by use of appropriate transducer frequency range and contact pressure we have been able to generate and detect waves with sensitivity to both shear and longitudinal mechanical properties. This was accomplished without the need of a special shear couplant, specimen geometry, or surface preparation.

Results of this study have clearly indicated that with increasing axial elastic modulus of the SiC/RBSN specimens, the velocity VL also increases. Conversely, loss of elastic modulus caused by oxidation of the fiber matrix/interface resulted in loss of velocity VL. These results are consistent with the theoretical predictions cited above. Furthermore, under the fabrication or heat treatment conditions in which the interfacial shear strength has increased, the velocity VS correspondingly increased; similarly, loss of interfacial shear strength resulted in decrease in velocity VS. This relationship has not been reported before with unidirectional fiber-reinforced composites.

We are not aware of a physical model that relates interfacial shear strength and shear velocity. However, it is possible that under the conditions in which we caused interfacial properties to change, the shear modulus of the fiber/matrix composite is also affected. Further work is needed to prove this concept and to better understand the

relationship between interfacial shear and shear velocity.

CONCLUSIONS

We demonstrated that for three important CMC parameters: density, SiC fiber type, and oxygen heat treatment; changes in the measured ultrasonic velocity VL correspond to changes in axial modulus E. Also, changes in the velocity VS correspond to changes in interfacial shear strength ISS. These results suggest that ultrasonic velocity can be used to measure and differentiate between two important strength parameters in ceramic matrix composites. These parameters typically are determined by destructive mechanical tests.

It is believed that the relation between VS and interfacial shear strength derives from the possible relationship between ISS and interfacial shear modulus in the specimens tested in our work.

Our work indicates qualitative relationships and sensitivities between ultrasonic velocity and key mechanical properties of ceramic matrix composites. To further realize the property prediction capabilities of the ultrasonic measurements, effort should be directed toward ultrasonic assessment of thermo-mechanical degradation and impact damage in ceramic matrix composites.

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TABLE I. - MECHANICAL AND ULTRASONIC DATA FOR SiC/RBSN COMPOSITES

Specimen	Density, g/cm^3	Axial modulus, GPa	Interfacial shear strength, MPa	Velocities, cm/μsec	
				VL	VS
As-fabricated SCS-6/RBSN	2.40±0.1	193±7	18±3[a]	0.756±0.033	0.385±0.015
HiPed SCS-6/RBSN	3.04±0.05	295±3	19±6[a]	0.851±0.45	0.383±0.045
As-fabricated SCS-0/RBSN	2.40±0.1	193±7	>200[b]	0.725±0.132	0.442±0.014
SCS-6/RBSN heat treated in oxygen at 600 °C for 100 hr	2.40±0.1	159±7	0.9±3[a]	0.549±0.32	0.275±0.011

[a]Measured by matrix crack spacing method.
[b]Measured by fiber push out method.

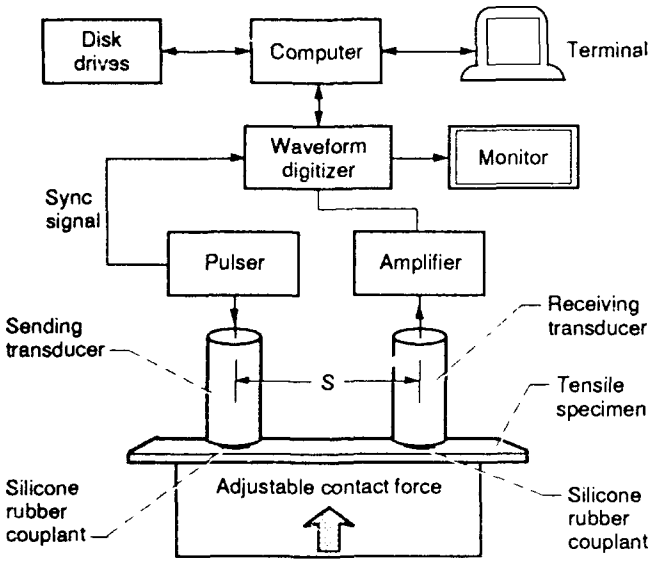

Figure 1.—Schematic of ultrasonic velocity measurement setup.

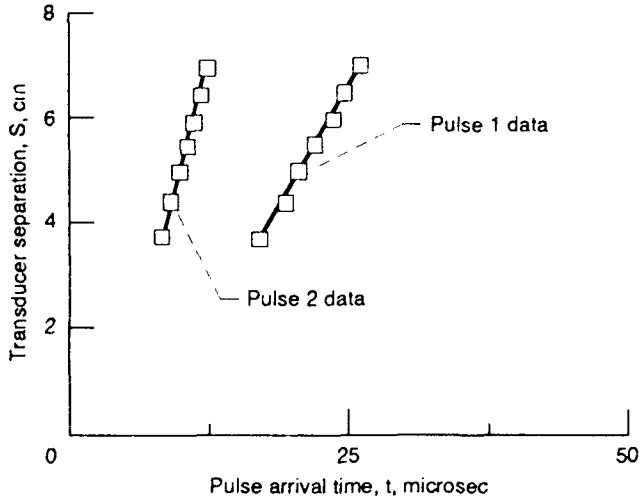

Figure 3.—Regression plots of spacing versus pulse arrival time for determining through-length velocity, $V = \Delta S/\Delta t$ i.e., the slopes of the curves.

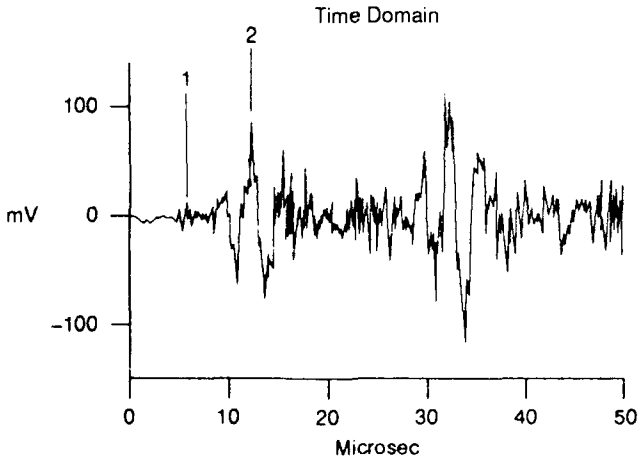

Figure 2.—Typical ultrasonic signal collected from a SiC/RBSN composite specimen indicating positions of the pulses 1 and 2 used to determine velocities VL and VS respectively.

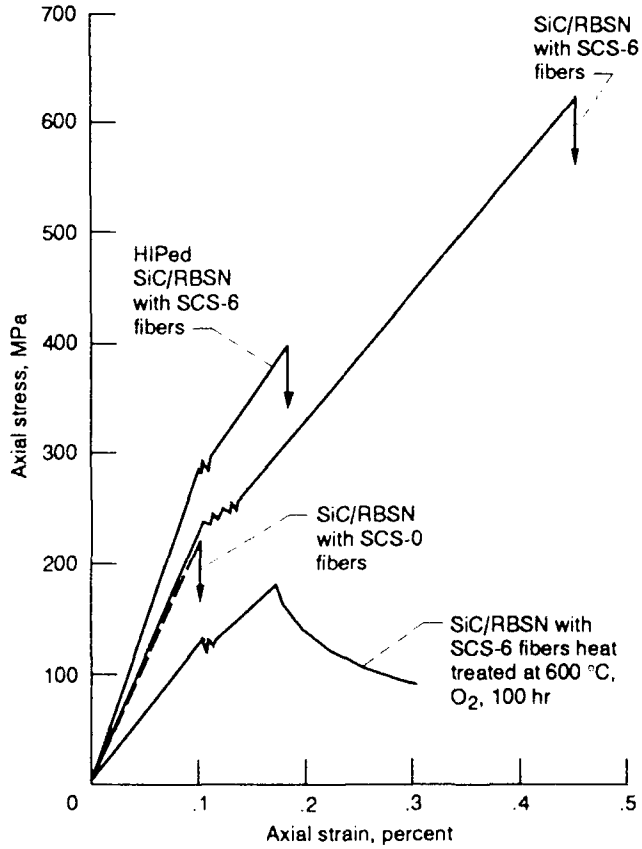

Figure 4.—Room temperature tensile stress-strain curves for SiC/RBSN composites.

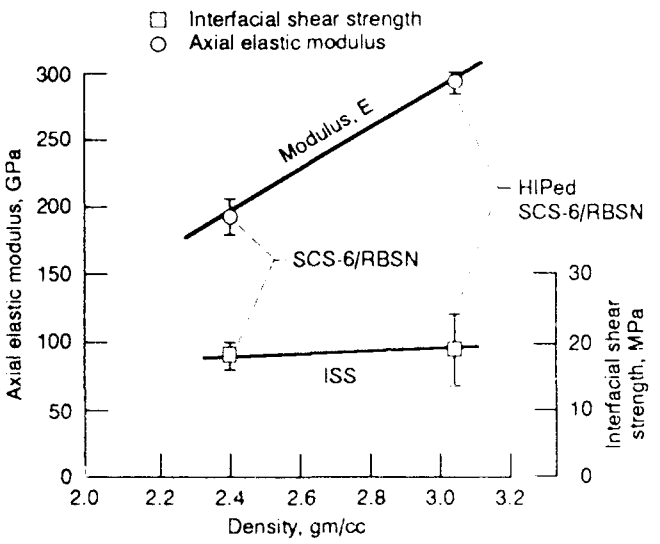

Figure 5.—Variation of axial modulus and interfacial shear strength with density for SCS-6/RBSN and HIP'ed SCS-6/RBSN composites.

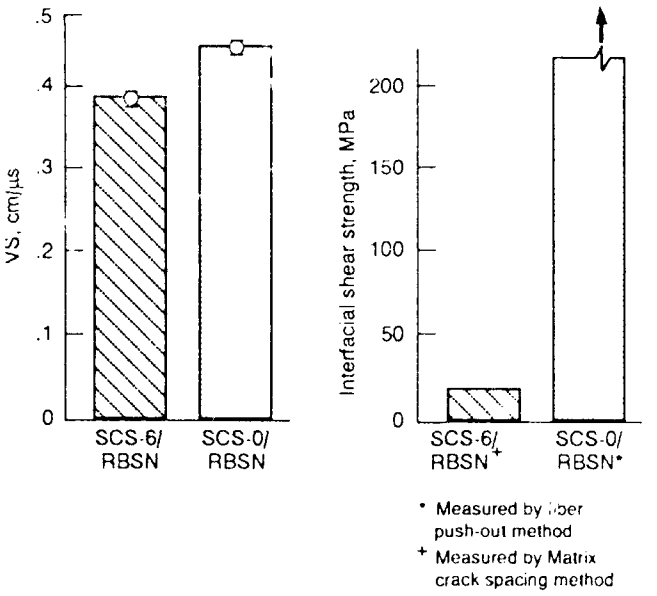

Figure 7.—Room temperature velocity VS and interfacial shear strength data for SCS-6/RBSN and SCS-0/RBSN composites.

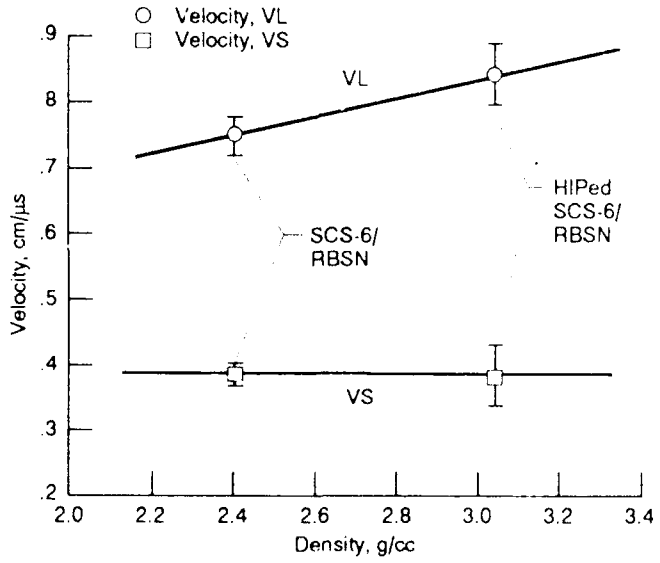

Figure 6.—Variation of velocities VL and VS with density for SCS-6/RBSN and HIP'ed SCS-6/RBSN composites.

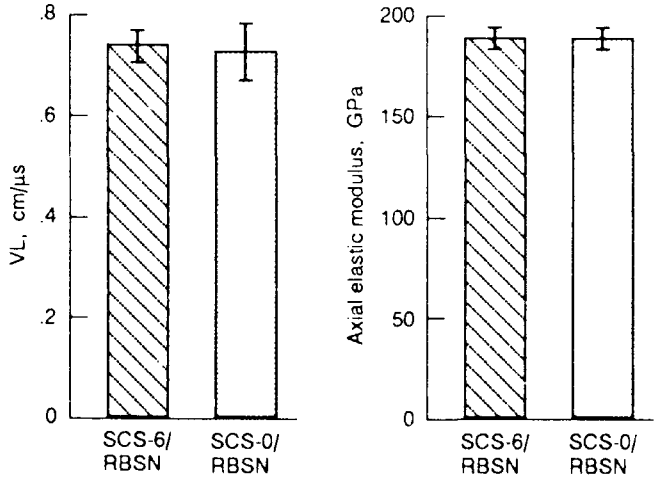

Figure 8.—Room-temperature velocity VL and axial modulus data for SCS-6/RBSN and SCS-0/RBSN composites.

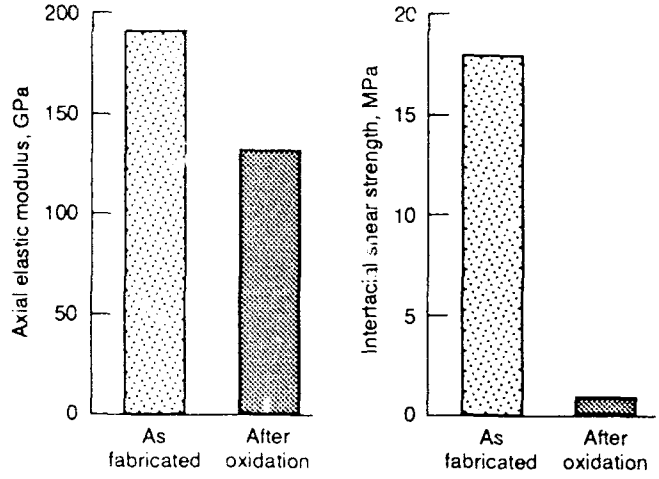

Figure 9.—Room temperature mechanical properties for SCS-6/RBSN composites before and after exposure in oxygen at 600 °C, 100 hours.

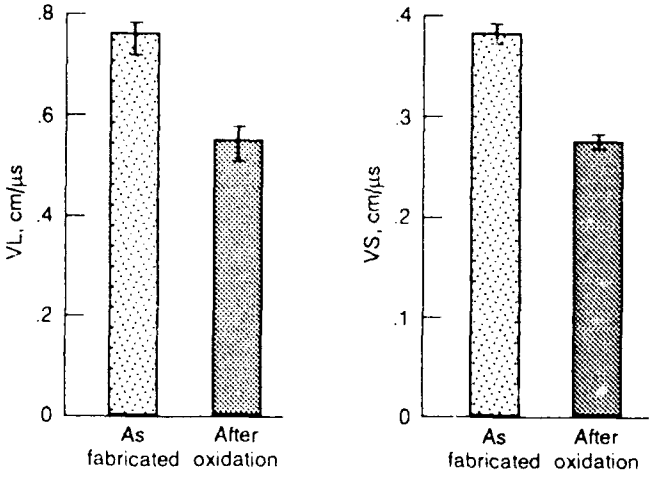

Figure 10.—Room-temperature velocities VL and VS for SCS-6/ RBSN composites before and after exposure in oxygen at 600 °C, 100 hours.

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16. Abstract
A novel technique for measuring ultrasonic velocity was used to monitor changes that occur during processing and heat treatment of a SiC/RBSM composite. Results indicated that correlations exist between the ultrasonic velocity data and elastic modulus and interfacial shear strength data determined from mechanical tests. The ultrasonic velocity data can differentiate strength. The advantages and potential of this NDE method for fiber reinforced ceramic matrix composite applications are discussed.

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